



DECLARATION UNDER 37 C.F.R § 1.132
OF GIOVANNI MANFRE, PhD

RECEIVED
NOV 21 2003
TC 1700

Sir:

I, Giovanni Manfre, PhD., do hereby declare and say as follows:

1. I received a Bachelor of Science degree (B.Sc.) in Physics and Mathematics from the University of Padova, Italy in 1963 and a Doctor of Philosophy in Material Sciences degree (Ph.D.) from the University of Surrey in 1966. I was employed from 1958 to 1993 in R&D in glass, polymer, composites, reinforcing and textile fibers, optical fibers, special metal processes, shape memory materials, rheocasting process, float and shaping glazing for automotive, responsible of R&D Fiat car makers of all transparent materials applications on cars, vehicles, train, airplane, ships, optical fibers, composite on cars for weight reduction, lubrication on engine, aerodynamic penetration. Since 1993 I have been acting as a consultant to:

- Isoclima Glass Transformers (Italy) in the areas of advanced armored glazing, multifunctional glasses as solar control, acoustic-thermal-electromagnetic radiation barriers, optics and special complex shape for cars, trucks, train, ships; switchable glasses (liquid crystals-electrochromics), anti-reflecting, water repellent (self cleaning), plastic glazing (polycarbonate, PMMA, PU), direct adhesive bonding and encapsulating of stratified and tempered glasses.
- Sekurit Saint Gobain (France) in the areas of switchable glasses, solar control direct coating, heatable glasses and IR (and UV) reflecting by metallized PET embedded in PVB and PU adhesives; dismantling and recycling glazing for ELV; flatness lighting integrated in glazing by LED and electro-luminescent (E) in vehicles, Electrochromic adhesives.
- Fiat and Pininfarina car manufacturers (Torino, Italy) in areas relating between glazing (windshield) mechanical and optical performances to avoid fracture (even by stones) and visibility discomfort. Heatable windscreen in symbiosis with IR reflecting to reduce the heating and air conditioner system in vehicles, Debonding for ELV recycling glazing.

- Bisazza (Italy) in the area of new mosaic for building applications and adhesive decorative panels in architecture.
- Isoclima-Schott Desag (Germany) in the innovative production of reflecting-heatable external mirrors.
- Wide Eye Co.(Bergamo, Italy) to develop a back mirror realized in plastics by injection moulding with wide angle of back vision (more than 45°) without image deformation and double image reflection.
- Debonding Ltd. (UK) to develop the debonding and bonding technologies by thermal expanded microspheres for easy replacement and ELV dismantling glazing and bonded panels in automotive industry and other fields.
- EEC Project (technical leader) for EC variable transmission adhesives for glass and transparent plastic glazing in automotive and building applications.
- Artech Publishing Int. Glass Technology (Milano, Italy) to produced 5 articles/year on glazing evolution. Report publications, patents and congress participation.

Additionally, I have delivered numerous lectures and authored and co-authored numerous articles and books in the areas of glazing technology. I have been a named author of more than 100 publications. In 2001, I received a GPD Award for having introduced the concept of all around visibility in vehicles contributing 1.5 m² of transparent surface. I am knowledgeable of the contents of the above-identified patent application and am a co-inventor.

2. With regard to the history of the present invention when, I as an inventor, started to approach the de-bonding technology, I had to face the following past experience of the problem:

- a. the debonding of any adhesive has to happen at command only when, for some reason, it is necessary to deactivate the adhesive (or any coating) from its substrate surface;
- b. the debonding agent activator must not interfere with the bonding process and the consistency of adhesive during its whole ageing operating life. In other words, the adhesive bonds at an interface with its substrate should not reduce their strength (commonly referred to in the art as lap shear) or function as a consequence of the ambient conditions such as temperature, humidity lights (UV, IR), mechanical fatigue and impact resistance. Conditions which can be summarised and controlled simply by any variation of the significant parameter "lap shear";
- c. the debonding action has to occur rapidly, at command and at a temperature levels that leave the adhesive structurally consistent so as to release it from a substrate at nearly zero load of lifting off (or peeling) and leaving the debonded surface as clean as possible;
- d. the debonding can occur in two ways: cohesive fracture of the adhesive or/and deactivating the bonds at interface with its substrate.

I also had to take into account that the strength required to deactivate the surface, by lifting off the adhesive or peeling, requires about 1/3 of the debonding energy compared with that of structure debonding by cohesion fracture.

Among the best adhesives (and coatings), in terms of strength and durability, are those on thermosetting polymers. Typically these are applied as a liquid mixture of low molecular weight monomers, these adhesives are wet and penetrate pores on the substrate surface. In some instances, insoluble products are formed that are mechanically interlocked and often covalently bound to the substrate to which they are applied. Examples of these conventional adhesives are polyurethane, epoxies, phenolics, melamines and the like.

These latter adhesives are essentially permanent and, as in the case of direct bonding the glazing with the vehicle frame. They bond with the frame from one side, and the black printing glass screen from the other side, by a special primer at the interface which has been found to avoid dangerous effraction of glazing during driving or car impact in certain conditions.

The technology employed to remove the adhesive from a substrate, is by cutting the cohesively attached adhesive with a wire using an applied force (usually carried out by a trained technician). However, this is a lengthy and costly operation which can result in damage to the trim of the car, fracturing the glass and damage to the surround by ambient powder debris. Some alternative processes have been tried over the last decade and these include, heating the adhesives with hot air, lasers, ultrasonics, IR, electrical current and so on. However, none have been found to abrogate the problems as they all create a degradation of the materials resulting in a char which is nearly impossible to easily debond even at temperatures above 180°C. Yet further approaches to debonding technologies are related to remove the adhesive by aggressive chemical agents applied to the adhesive surface so as to degrade them at the interface. Other electrochemical or memory effect activator and so on are have also been found ineffective.

In conclusion the cited references do not provide for the material of an adhesive or interface to sudden and readily cleave from the substrate. Methods to date still require conditions that are time and labour intensive as well as being quite harsh and likely to cause damage to the underlaying surface.

In summary, there is no teaching in the reference to a **sudden debonding** cleavage. Nor is there any direction to use a different technology from the present bonding technology in automotive industry which can sustain the mechanical features of an adhesive so that it does not deteriorate even after 15 years of ageing, with the debonding activators dormant over that period but with the ability to make them

suddenly operational at command for glass displacement and for end life vehicles (ELV) regulations for recycling.

In other words, the debonding technology of the present invention had to be approached with the limits of certain bonding process and materials with parameters intrinsically able to give an industrial feasibility as a result of a project.

After several initial studies with alternative activators and process, I found the thermoexpandable microcapsules (TEMs) having certain characteristics, seemed to fit the above requisites. The present patent application was filed as the consequence of the experimental results with the microcapsules mixed in the adhesive matrix and/or in the primer but always with the aim to debond the adhesive bonded surface at interface and not to fracture the cohesive structure of the adhesive itself. The present invention meets the need of the glazing industry in the provision of a composition to which the debonding technology with the parameters mentioned hereinbefore could be applied.

3. With regard to the Office Action, point by point, I make the following statements based on the quotation of 35 USC 103 (a) in order to established a background of the inventive step summarized as follows:

- 1) determining the scope of the cited reference;
- 2) ascertain the differences between the cited reference and the claims issue;
- 3) resolving the level of ordinary skill in the pertinent art;
- 4) considering objective evidence present in the application indicating obviousness and non obviousness.

Hartman et al (US 5,476,712)

- 1) Hartman et al. illustrates an adhesive tape providing a desired compressibility and tear resistance characteristics, useful in mounting it on flexographic printing plates. The core of the tape is a closed cell foamed layer between at

least one skin. The foamed core layer comprises a thermoplastic (not thermosetting) elastomer matrix containing a distribution of voids, provided by the thermally blowing agent which expand at elevated temperature upon heating to form a cellular structure. The blowing agent temperature matches the extrusion temperature of the coextrusion process to realise the compressibility properties of the core (principal part of the invention) of the multi-layer tape, able to adhere to the cylinder of a printing process and so to enable an easy continuous removal of the tape from the cylinder moving surface. The selection of the materials for matrix and the blowing agent has been selected to have adhesive layers coextruded and the foamed cellular core being compressible due to voids in a thermoplastic elastomer and thus to simplify the continuity of the process removal of the tape from the cylinder during the printing.

- 2) The differences between this reference and the present invention are as follows:
- the expandable beads (see col. 4 lines 5-20) produce a cellular construction provided by an elastomeric materials and voids substantially distributed uniformly through the matrix and which does not extend them into skin layer. The voids are formed by expanded thermoplastic shell microsphere containing the vapour phase of an organic liquid which evaporates during the thermal blowing and expanding step, during the coextrusion process to realise the tape. Hartman et al use Expancel® (column 6 line 65);
 - the present invention uses microspheres that do not produce voids but have to be still present in an unexpanded state in the final product and which do not degrade the strong structure of the adhesive thermosetting materials for more than 10 years (the microbeads remained unexpanded in the adhesive up to a command heating). At the end of its use the sudden expansion process does not produce voids by expanded microbeads but at the **surface of the adhesive interface** they create (no unuseful voids) pressure actuators to debond physically the adhesive surface on the substrate. The foamed voids do not produce debonding in any way at the surface and if they are produced, even on

the surface, at command (which is not the purpose of the Hartman patent) they cannot be debonding actuators but can only participate in surface degradation.

- In conclusion the reference has a completely different purpose (compressible layer not debonding layers), it provides voids and not debonding actuators, the voids are produced during the process not at the end use of the product, they degrade the cohesive structure not the surface adhesion. As a consequence there is no evidence that the reference can produce the same results of the present invention or vice versa.

- 3) 4) Even the level of ordinary skill cannot be considered pertinent art as the debonding composition of the present invention requires microspheres containing a blowing agent in such a way that the expanded microspheres, remain still integral in order to be activated rapidly and at command for surface adhesive debonding which is the main purpose of the present invention. There is no teaching to provide the microspheres of the present invention which may remain dormant for more than 10 years ageing without interfering with the structural strength of the adhesive.

Everaerts et al (US 5,695,837)

- 1) The scope and contents of the reference is to realise a foam-like pressure adhesive tape including also as expanded as unexpanded (Expancel® and Micrpearl® column 10 line 35 and 36) microspheres in order primarily to reduce the density by nearly 100% of the thermally expanded microsphere mentioned in the reference. The density reduction is increased by mixing other kinds of commercially available glass and ceramics microspheres. This reference is pertinent for thermoplastic materials not for thermosetting. The adhesive of Everaerts (see col. 10 lines 55-65) is useful for bonding to a variety of different surface including low energy surfaces and is indicated as useful for paints adhesion of 11N/dm in 90° peel adhesion.

- 2) The expanded microspheres of Everaerts are used for **foaming** the adhesion. There is no direction nor mention of their presence for their use in decreasing adhesion at interface with their substrate, suddenly nor after at least 10 years of their being in a dormant state without degrading the structure of the strong adhesion, cohesive in volume or adhesive at the surface. Accordingly the microspheres of the present invention are not obvious with respect to this citation.
- 3) 4) There are no indications in the reference to provide the microspheres (ordinary market microspheres) as a debonding activator at the interface. Accordingly, even with ordinary skill, the present invention is not obvious over the citation.

Makhlouf (US 5,712,317)

- 1) The aim of this reference is to reinforce thin rigid plate by a curable composition onto one of its sides, using a composition comprising a thermosetting polymer and expandable microspheres (Expancel® column 3 line 61) mixed with other reinforcing materials as glass fibers. The present available thermally expanding microspheres is simply used to prevent shrinkage of the composition during cure in order to retain the shape and appearance of the thin rigid plate. The expandable microbeads are advantageous (col. 3 lines 45-55) in that they do not cause the composition to fall or blow off the surface unlike foaming agents.
- 2) As an obvious consequence, Makhlouf experimented that his use of expandable microspheres prevents the deactivation of the composition from the thin plate during curing and assuring that certainly no silent microspheres are present to produce debonding which is exactly the contrary to the present invention and our debonding technology.

- 3) 4) There is no obvious tentative and no factual comparative evidence that the debonding patented microspheres: in its size and their distribution, shell thickness, blowing agent, temperature of expanding range, time of the expansion, their uniform mixing have all to be invented to activate the debonding strength rather to prevent the deactivation as Makhoulouf declare in its claims. The present invention does not only mention the microbeads (Claims 1-23) as pressure deactivating activators but also their participation in the adhesive interface or in the primer (Claims 1 and 24) in order to avoid the bonding "silent" effect like those of Makhoulouf.

Gehlsen et al (US 6,103,152)

- 1) In this citation, again the temperature expandable microspheres (Expancel® column 8 lines 31 to 32) are used to create, in a die extrusion process, conditions to decrease the density (up to 50%) of the extruded product and especially to reduce the weight of shoes. The choice of the microspheres has also to take into account the smoothness of the extruded materials which is certainly the intrinsic main difference from other present and normal use of microbeads. This citation teaches microbeads for density reduction and surface smoothness.
- 2) 3) 4) The differences are evident, there is no teaching in this citation to provide the microspheres of the present invention and to mix them with adhesives so as to obtain an instant debonding result at command.

EP 0717091

- 1) EP 0717091 discloses a foamed pressure sensitive adhesive by a mix of thermoplastic polymers, a tackifying resin, an isocyanate a back home polymers, 2 active hydrogens able to react with isocyanate and finally expandable particulate materials. The expandable particulates (Expancel® page 5 line 52, 54, page 7 line 26) provide a density that is less than about

40% of the theoretical density. The compression tends to irreversibly deform the produced adhesive due to adherence between opposite side of the cells in the cellular structure.

- 2) The particle material of the citation is in effect thermally expandable microbeads, easily available on the market (Expancel™ and Microperl™) which are normally used to reduce the density of the materials or to increase the pressure foam sensitive adhesive but this use does not obviously imply any sudden deactivation properties. It is not obvious to apply the microbeads on the reverse attitude to physically create debonding at interface rather than a sensitive compressive adhesion. A normal skilled artisan cannot deduce the microspheres of the present invention and their activation at command which can operate only at an interface and after a certain ageing time. Density reduction and foam adhesive compression is, at present, a normal and known use of the expandable microbeads, however deactivation action has not been considered at all in the reference.

- 3) 4) There is no direction in this citation to debonding; certainly on the the objective evidence, even experimentally verified by anybody on the field of foam adhesives, this citation is not pertinent to adhesive bonding at an interface or to physical debonding as described in the present application.

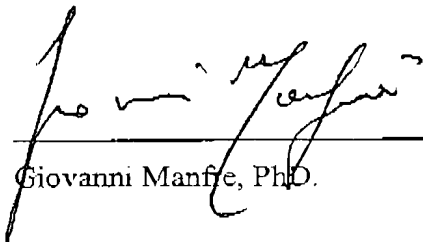
4. I hereby declare that the debonding technology achieved by expandable microbeads of the present invention compared with the foam technology, although they use microbeads, is not obvious in view of the cited references. Microbeads used in the reference are present only to reduce the density and not to create bonding at an interface. In the normal skill of reference no concept and process can be deduced for their use as a debonding activator at interface where the alternative concept of voids does not orient on debonding the adhesive from the substrate but only to create a sensitive compressive adhesion. Thus, the present invention should be considered non-obvious over the cited references as it would not be possible to apply cited reference microspheres to a composition for debonding technology. Moreover the

cited references have not recognised the concept of the presently claimed application, neither in process, theory nor by experiment.

5. I hereby provide Exhibit A which shows experimental data comparing the reference microbeads Expancel® and Micropearl® with those of the present invention. Additionally we provide for the Examiner's viewing a CD-ROM of a "real time" experiment illustrating the different rates and extent of expansion of the cited reference microbeads with those of the present invention.

6. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

In re: Bain, et al.
Serial No.: 09/980,219
Filed: April 18, 2002



Giovanni Manfre, PhD.

13. NOVEMBER. 2003
Date

P32052us.dec